RESTEK
Chromatography Products

Environmental Applications

Techniques for Optimizing GC Analysis of Ethylene Glycol in Water

The analysis of ethylene glycol in water is a very common test in environmental laboratories. Many of these samples originate from water runoff at airports, where ethylene glycol is used as a de-icing agent for airplanes during winter months. Because ethylene glycol is highly soluble in water, it is not easily concentrated by purge and trap. Therefore, the most frequently used sample introduction technique is direct aqueous injection. The direct aqueous injection of ethylene glycol can be challenging because, if not done properly, it can be difficult to attain reproducibility and good peak shape. The large expansion volume of water can cause backflash, carryover can cause inconsistent results, and excess water can extinguish the FID flame. These problems can prevent achieving the detection limit for ethylene glycol, which may vary in the 1-10 ppm range.

Poor Peak Shape

With a column head pressure of 10 psig and an injection port temperature of 250 °C, a 1 µL injection of water will expand to 1,420 µL of vapor. This large vapor cloud exceeds the volume of most inlet liners, causing backflash. If backflash occurs, the vapor cloud can expand out of the liner and injection port and result in poor sample transfer onto the column. Also, the glycol compounds are not focused in a narrow band but, instead, are focused in the condensed water that beads onto the column walls, so the compounds of interest can elute as split peaks. This peak splitting effect is most apparent when performing a splitless injection because of the solvent focusing required. Split peaks and backflash compromise the analysis by causing irreproducible peak shapes.

One technique to reduce the effect of vapor expansion and poor solvent focusing is the use of a Uniliner® or Drilled Uniliner® for the injection port liner. This liner forms a leak-free connection with the column end (Figure 1), thereby ensuring a complete sample transfer. The column sealed at the bottom of the liner minimizes sample backflash, eliminating the potential for ghost peaks. By using a Uniliner® liner, the aqueous ethylene glycol sample is vaporized and quantitatively transferred to the column in a focused band, thereby achieving reproducible peak areas. Uniliner® liners are available for conversion of packed column injection systems and for split/splitless injection systems.

Sample Residue Carryover

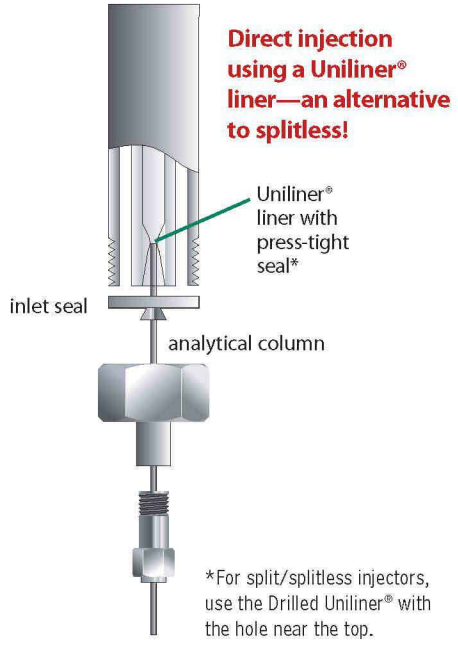
Carryover is another problem associated with ethylene glycol analysis. When analyzing glycols, carryover can be caused by sample residue in the syringe being carried over from one injection to another. If the syringe is not properly cleaned between analyses, carryover will cause inconsistent results. It was determined that rinsing the syringe with either water or water:methanol (50:50) three to six times between each injection will eliminate sample residue and minimize the possibility of carryover.

FID Flameout

Column stationary phase choice is a critical consideration when analyzing glycols in water via direct injection. Water analyzed on a nonpolar stationary phase, such as the Rtx®-1 column, or on a moderately polar stationary phase, such as the Rtx®-200 column, could cause the flame on the FID to be extinguished. This is because the water will not partition properly and will "bead up" on the phase, producing a large plug of water that passes through the detector and extinguishes the flame.

To minimize the possibility of extinguishing the flame, select a polar stationary phase that is more compatible with water. The Stabilwax® stationary

Figure 1 The Uniliner® inlet liner forms a leak-free connection, minimizes backflash, and helps transfer the sample from inlet to column.



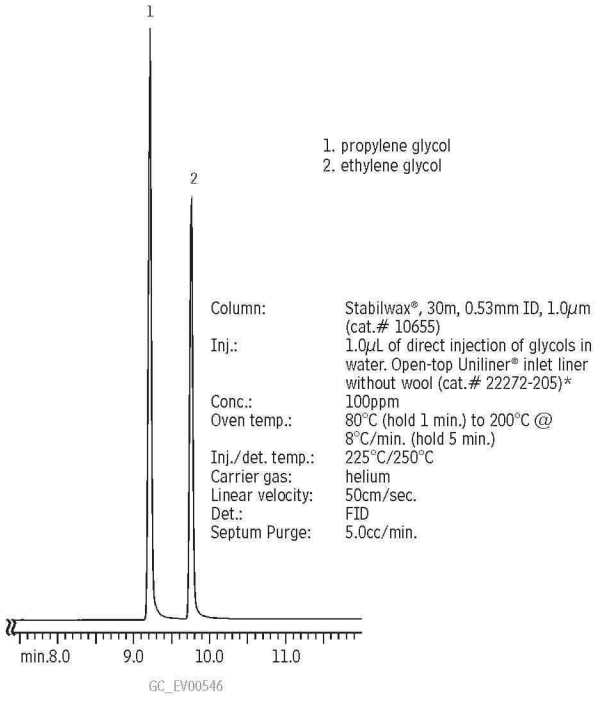
phase is one of the more polar phases, making it a good choice for water injections. It allows water to partition properly, which prevents it from beading up on the stationary phase and quenching the FID flame.

The Stabilwax® column can easily handle direct aqueous injections without showing any signs of degradation. Testing of the Stabilwax® column was performed by injecting 1 µL of a water standard 100 times. Peak shape and response of ethylene glycol and propylene glycol remained consistent throughout the analyses (Figures 2 and 3). The Stabilwax® column also allows sensitive detection of low ppm-levels of glycol compounds. Notice the 5 ppm standard for ethylene glycol in water is easily achieved, and peak shape is maintained when compared to a 25 ppm standard (Figure 4).

Conclusion

You can achieve better response and reproducibility for the GC analysis of ethylene glycol in water by using direct injection with a Uniliner® liner, a polar capillary column such as Stabilwax®, and multiple syringe washes between runs. Using these techniques can assist in attaining reproducible analyses with detection limits in the low ppm range.

Figure 2 The Stabilwax® column shows good peak shape and response for ethylene glycol after 100 injections.



*For split/splitless injectors, use the Drilled Uniliner® with the hole near the top.

Figure 3 The Stabilwax® column combined with a Uniliner® liner shows remarkable response consistency.

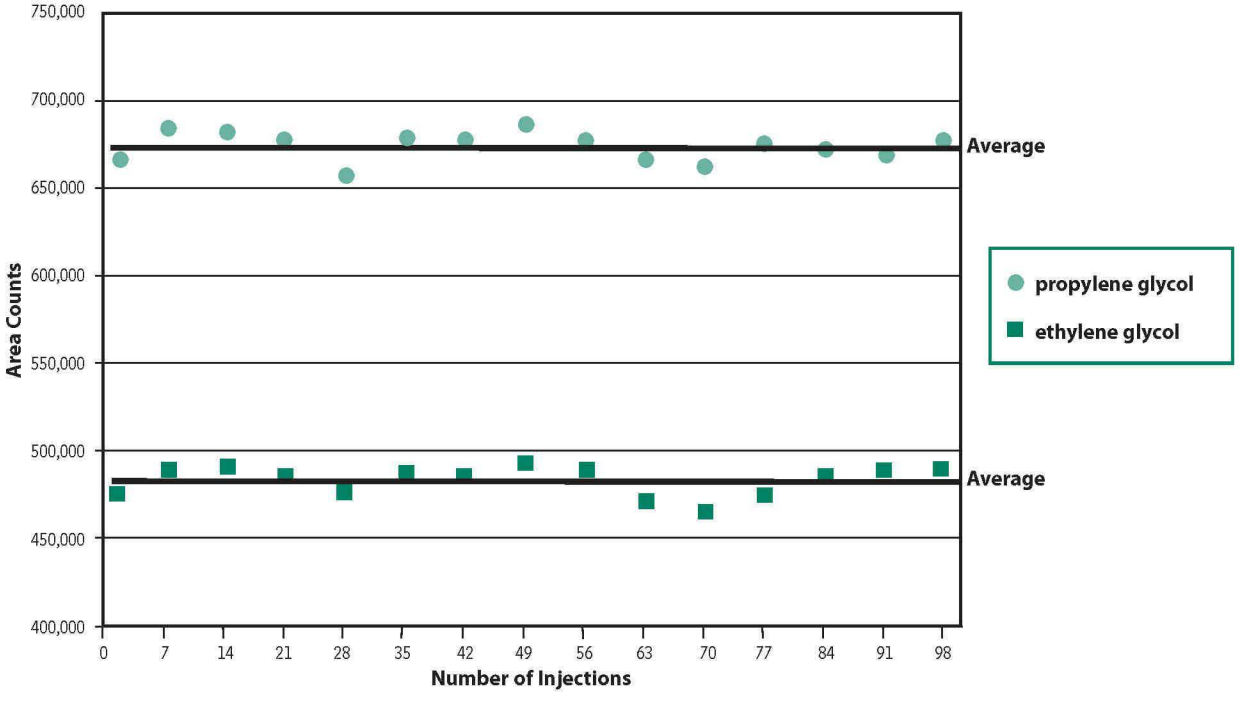
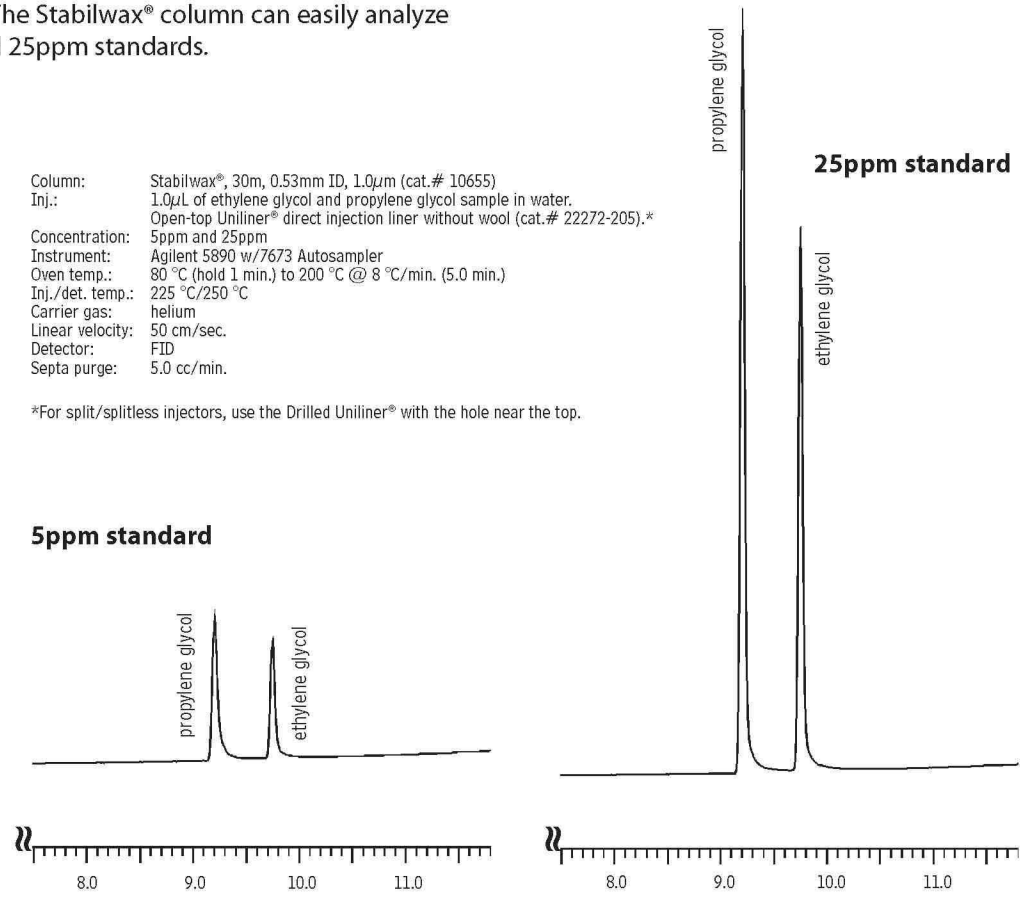


Figure 4 The Stabilwax® column can easily analyze 5ppm and 25ppm standards.

Column: Stabilwax®, 30m, 0.53mm ID, 1.0µm (cat.# 10655)
Inj.: 1.0µL of ethylene glycol and propylene glycol sample in water.
Open-top Uniliner® direct injection liner without wool (cat.# 22272-205).*
Concentration: 5ppm and 25ppm
Instrument: Agilent 5890 w/7673 Autosampler
Oven temp.: 80 °C (hold 1 min.) to 200 °C @ 8 °C/min. (5.0 min.)
Inj./det. temp.: 225 °C/250 °C
Carrier gas: helium
Linear velocity: 50 cm/sec.
Detector: FID
Septa purge: 5.0 cc/min.

*For split/splitless injectors, use the Drilled Uniliner® with the hole near the top.



Stabilwax® Columns (fused silica)
(polar phase; Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	1.00	40 to 240/260°C	30-Meter	10654
0.53mm	1.00	40 to 240/260°C	30-Meter	10655

Excellent peak shape for glycols produces lower detection limits.

Why let a small leak turn into a costly repair?

Protect your data and analytical column by using a **Restek Leak Detector**.

Restek Electronic Leak Detector
Features & benefits include:

- Optimized sample flow path.
- New ergonomic, hand-held design.
- Rugged side grips for added durability.
- Handy probe storage for cleanliness and convenience.
- Longer lasting battery, up to 6 hours of continuous use.
- Automatic shut-off.
- A convenient carrying and storage case.
- Easy to clean probe assembly.
- A universal charger set (US, European, UK, and Australian plugs included).

Description	qty.	cat.#
Leak Detector with Hard-Sided Carrying Case and Universal Charger Set (US, UK, European, Australian)	ea.	22839
Soft-Side Storage Case	ea.	22657
Small Probe Adaptor	ea.	22658

Avoid using liquid leak detectors on a GC! Liquids can be drawn into the system.

Backed by a 1-year warranty, Restek's Leak Detector sets an industry standard for performance and affordability in hand-held leak detectors.

Caution: The Restek Electronic Leak Detector is designed to detect trace amounts of hydrogen in a noncombustible environment. It is NOT designed for determining leaks in a combustible environment. A combustible gas detector should be used for determining combustible gas leaks under any condition. The Restek Electronic Leak Detector may be used for determining trace amounts of hydrogen in a GC environment only.

Restek Liners—ensure complete sample transfer and eliminate effects of backflash.

Liners and Inlet Adaptor for Agilent Packed Column Injectors

COLUMN INSTALLS THIS END

DI Liners for Agilent GCs** (For 0.25/0.32/0.53mm ID Columns)	Benefits/Uses	ID* OD x Length	Similar to Agilent part #	ea.	cat.# 5-pk.	25-pk.
 Drilled Uniliner (hole near top)	trace, active samples, high recovery & linearity	4.0mm 6.3mm x 78.5mm	—	21054	21055	20998
 Drilled Uniliner (hole near bottom)	trace, active samples, high recovery & linearity	4.0mm 6.3mm x 78.5mm	G1544-80730	20756	20771	—
 Double Gooseneck Drilled Uniliner (hole near top)	trace, active samples, high recovery & linearity	4.0mm 6.3mm x 78.5mm	—	20508	20509	—
 Double Gooseneck Drilled Uniliner (hole near bottom)	trace, active samples, high recovery & linearity	4.0mm 6.3mm x 78.5mm	G1544-80700	20954	20989	—
 Drilled Cyclo-Uniliner (hole near top)	trace, dirty, high MW, active samples, high recovery & linearity	4.0mm 6.3mm x 78.5mm	—	22979	22980	—

*Nominal ID at syringe needle expulsion point.
**Hole in Drilled Uniliner liner makes direct injection possible with EPC-equipped Agilent 6890 & 7890 GCs!

Uniliner® Liner

for 1/4-Inch Packed Injection Port Conversion

- Reduces solvent tailing.
- Versatile—0.53mm ID version can be used in the direct (DI) or on-column (OC) injection mode.
- Incorporates a gentle taper that seals the column and reduces dead volume in direct injection mode.
- Available in various designs.

did you know?

On-column injections can be performed only with 0.53mm ID columns because 26-gauge needles do not fit into the bore of 0.32mm ID columns, or into the Uniliner® liner taper.

Description	Column ID Injection Mode*	ea. cat.#	5-pk. cat.#
Uniliner Liner (small buffer volume chamber 60mm long, for injections ≤2µL)	0.53mm DI or OC	20902	20903
Uniliner Liner (large buffer volume chamber 85mm long, for injections ≤4µL)	0.32 & 0.53mm DI only	20308	20309
	0.53mm DI or OC	20301	20305
Cyclo-Uniliner Liner (for active, dirty samples)	0.32 & 0.53mm DI only	20319	20320
Open-Top Uniliner Liner (packed with wool)	0.32 & 0.53mm DI only	20315	20316
Low Volume/Purge & Trap Uniliner Liner (1mm ID x 5mm OD: use in 1/4" injection ports to troubleshoot purge & trap units)	0.32 & 0.53mm DI only	20307	20314
Uniliner Liner Adaptor (required for installing Uniliner liners in 1/4" injection ports) 	Includes 1/4-inch nut & graphite ferrule, 1/16-inch nut, and 0.8mm ID graphite ferrule.	Stainless Steel	Siltek-Treated
	For injection ports <8cm	20310	22282
	For injection ports 8-15cm	20311	
1/4" Ferrules for Uniliner Liner Adaptor	cat.# 20234 (5-pk.)		

*DI = direct injection, OC = on-column injection

Note: a Uniliner® inlet liner must be used with a Uniliner® Liner Adaptor (cat.# 20310 or 20311) for 1/4-inch injection ports. Remember to include a liner adaptor when ordering a Uniliner® liner, unless you are purchasing replacement Uniliner® liners.

RESTEK

Lit. Cat.# EVAN1248-INT

Restek U.S. • 110 Benner Circle • Bellefonte, PA 16823 • 814-353-1300 • 800-356-1688 • fax: 814-353-1309 • **www.restek.com**
Restek France • phone: +33 (0)1 60 78 32 10 • fax: +33 (0)1 60 78 70 90 • e-mail: restek@restekfrance.fr
Restek GmbH • phone: +49 (0)6172 2797 0 • fax: +49 (0)6172 2797 77 • e-mail: info@restekgmbh.de
Restek Ireland • phone: +44 (0)2890 814576 • fax: +44 (0)2890 814576 • e-mail: restekeurope@aol.com
Restek Japan • phone: +81 (3)6459 0025 • fax: +81 (3)6459 0025 • e-mail: ryosei.kanaguchi@restek.com
Thames Restek U.K. LTD • phone: +44 (0)1494 563377 • fax: +44 (0)1494 564990 • e-mail: sales@thamesrestek.co.uk

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